# THE INSTITUTE SPOKESMAN

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## New Members . . .

Again this month "The Institute Spokesman" is delighted indeed to extend a warm and cordial welcome to three new members of the National Lubricating Grease Institute.

Joining as an Active member and designating Mr. W. R. Gaylor to maintain contact between our two organizations is the Standard Oil Company (Kentucky) whose offices are in the Starks Bldg. at Louisville.

Since 1886, when its mule drawn tank wagons were a familiar sight on muddy southern roads, the Standard Oil Company of Kentucky has been one of the South's leading distributors and marketers of petroleum products.

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Ever alert to the increasing needs of a progressive new South, Standard Oil of Kentucky is continuing to expand its service and operating facilities — to match the strides of a "Dixie On The March".

One Associate Member has been added: The Harshaw Chemical Company, 1945 East 97th Street, Cleveland 6, Ohio, whose Assistant Secretary-Treasurer, Mr. A. D. Perry, will serve as liaison.

The Harshaw Chemical Company, which was founded more than a half century ago, manufactures upwards of 1,500 different chemicals primarily for industrial consumption. For many years, the Company has been manufacturing for the grease industry a wide variety of metallic soaps, particularly sterates, laurates, palmitates, and lead napthenates for hypoid lubricants, all of which have been the result of an aggressive research program. Some of the other principal products manufactured by Harshaw are catalysts for the petroleum industry, chemicals and anodes for many types of electroplating; a wide variety of fluorides; anhydrous and aqueous hydrofluoric acid; cobalt, manganese and lead driers for the paint, varnish, linoleum and printing ink industries; refined glycerine; opacifiers, and an extensive line of colors and chemicals for the ceramic industry; special glazes and porcelain enamel frits; synthetic optical crytals; and fungicides.

To Technical Membership in the Institute we welcome the Mellon Institute of Industrial Research, University of Pittsburgh, Pittsburgh 13, Pa. Mr. C. Malcomb Findlayson will provide the point of contact between the two Institutes.

Mellon Institute is an endowed nonprofit institution, a corporate body, for research in the pure and applied natural sciences, for training research workers, affording them facilities for special development, and for providing techni information adaptable to public adv tage.

Mellon Institute grew out of a p conceived by Robert Kennedy Dunc in 1906 to make scientific research m available to the public and applic more effectively to the Industries. procedure referred to as the Indust Fellowship System was evolved wh gave a manufacturer the privilege establishing a temporary fellowship a university for the investigation o particular problem, the solution of wh would mutually benefit both the ma facturer and the public. Duncan initia this system at the University of Ka in 1907, and in 1910 Andrew W. Mel and Richard B. Mellon called him put it into practice at the University Pittsburgh.

The idea was accepted with inter by the industries and in 1913 it w thought to have advanced far enough deserve a permanent organization a name. The Institute remained a part the University of Pittsburgh until 192 when it was separately incorporated. T Institute cooperates with the Univers and the Junior members of its resear


### *The* INSTITUTE SPOKESMAN

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staff may enjoy the opportunities of graduate students there. However, the researches of fellowships are of post-doctoral character, with the fellows and their aides having the status of salaried workers.

The arrangement is as follows: A manufacturer seeking to investigate a problem, or hoping for general benefit from a research program, donates money for a temporary fellowship at the Institute. A fellowship agreement is drawn up between the Institute and the donor setting forth the purpose and the terms. The fellow selected must be acceptable both to the donor and the Institute. Eighty fellowships are now in operation, some of which have been renewed over a period of 30 years.

The donor has control over the research findings of the fellowship it supports and over their patenting and publication. The fellows often find later employment with their donors. The Institute defrays the overhead expenses not chargeable to a particular donor and the cost of equipment of general long-term use. Thus, the Institute provides facilities for researches which if conducted individually would be much more costly. In addition, the physical separation of the Institute and its laboratories from production plants is regarded as a great advantage to long-range company research.

It is nice to have these additions to the N.L.G.I. Membership which marks the start of a mutually pleasant and constructive association.

These new members, along with older ones will be making the contribution to the activities and accomplishments of the organization.

## N.L.G.I. Board Meets

President H. P. Hobart has called a meeting of the N.L.G.I. Board of Directors for 10:00 A.M. on Friday morning, September 19th, 1947 at the Hotel Traymore, Atlantic City, New Jersey. Important matters for the consideration of the Board are on the agenda for this meeting, the final arrangements for the 15th Annual N.L.G.I. Convention scheduled for the Edgewater Beach Hotel, Chicago, October 16th-18th, 1947, election of a new director to fill the unexpired term of Mr. L. C. Welch, retired, the report of the Finance Committee concerning the budget for 1947-48. Present indications are that the Board meeting will be well attended.

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# *Evaluation of* WATER RESISTANCE PROPERTIES *of Lubricating Greases*

• **By T. G. Roehner and E. S. Carmichael,**
***Socony-Vacuum Laboratories Technical Service Department***

**About the Author**

*Mr. T. G. Roehner was born in Brooklyn, New York, in 1900. He received his Bachelor of Chemistry Degree from Cornell University in 1924. For six years immediately following graduation, Mr. Roehner worked in Rancagua, Chile, South America, with the Braden Copper Company, a subsidiary of Kennecott Copper Company. His starting position was that of chemist, from which he rapidly advanced to a test engineer and finally Chief Chemist. In 1930 the author started with the Standard Oil Company of Brooklyn, New York, as a chemist and became supervisor of Product Development. His present position is that of Technical Director of the Technical Service Laboratory, Socony-Vacuum Oil Company, Inc., at 412 Green Point Avenue, Brooklyn, 22, N.Y. Mr. Roehner speaks with the authority of a background of sixteen years experience with grease problems from all angles — analytical, formulation, laboratory evaluation, manufacturing and servicing under actual service conditions.*

*T. G. Roehner, Technical Director, Technical Service Laboratories, Socony-Vacuum Oil Co., Inc.*

The term "water resistance" is often loosely applied when describing the properties which a lubricating grease should have, in order to give satisfactory performance where exposure to water will occur. It is true that the ability of the grease to resist wash-out from the bearings and its corrosion prevention characteristics are both related to water resistance properties, but confusion in recommendations may be avoided if recognition is given to the fact that the grease which resists wash-out best may not also satisfactorily protect the bearings from serious rusting. For both of these requirements it is not unusual to find that recommendations are based primarily on the metallic radical of the soap component of the grease. Calcium, aluminum, lithium and barium are regarded as inherently resistant to water, while sodium is avoided where that characteristic is desired. Selection of greases on that basis will not consistently assure satisfactory products, because experience has demonstrated that other components of the lubricants may possibly have equal or more important effects.

A major difficulty in evaluating the relative effects of each of the various factors involved is the lack of agreement regarding laboratory test methods and their limitations. Products which show outstanding performance in certain tests have been failures and vice versa when used under actual service conditions.

From a broad viewpoint, the test data have been misleading in some instances because sufficient allowance has not been made for the relative importance of so-called water repellency and water absorption characteristics. The former may be the determining factor when resistance to wash-off is of interest, or, in other words, when water flows over the grease at a rate which will not remove the lubricant by physical force alone. Water absorption properties is a loose term applied to those characteristics which are the predominating factor when resistance to wash-out from the bearings is of interest. Under service conditions, water repellency properties are a function of the ability of the excess grease, particularly that adhering to the outside of the bearing, to act as a seal preventing the entrance of water in the bearing. After the water is entrapped in the bearing and is worked into the grease by movement of the bearing, water absorption properties come into play as they, by definition at least, determine how much water will be retained, and with what effect on the structure of the grease and its ability to stay put.

Resistance to wash-off is often determined by using plate immersion or dip tests. Samples are smeared uniformly over plates and observations and/or loss in weights are noted after the coated plates have been dipped in the test water under prescribed conditions. In this type of test, conventional calcium, aluminum and lithium base greases, for example, will show good repellency while conventional sponge greases made with soda tallow soap will wash off quite rapidly.

The methods employed for evaluation of resistance to wash-out provide for working water into the grease. Probably the two best known methods of this type are those included in certain Army Navy Aeronautical Board Specifications, i.e., The Water Absorption Test described in AN-G-3a and AN-G-10 and The Water Resistance Test given in AN-G-5a, AN-G-15 and AN-G-25.

The former was developed by the Naval Engineering Experiment Station at Annapolis, Maryland. The test equipment consists of a vertical steel shaft having a mushroom head 1-7/16″ in diameter and ¼″ thick attached in a vertical position to an electric motor rotating at about 1200 r.p.m. Twenty grams of the grease sample are placed in a cylindrical flat dish 2¾″ in diameter and 1½″ deep. Then distilled water is added to the grease in 1 ml. portions. The grease is worked between each addition of 1 ml. of water until inspection shows that no further water will be taken up. Each ml. of water is equivalent to 5% of water. The amount of working which the sample receives is quite drastic to and is intended to be equivalent to that which the grease would be subjected in a bearing in service.

The Water Resistance Test may be best described by quoting the procedure given in AN-G-15.

"A No. 204K Conrad type ball-bearing shall be packed with 4.0 grams of grease and clamped in a tight fitting housing which allows the inner race to turn freely, mounted on a horizontal shaft, and rotated at 600 r.p.m. A fine stream of distilled water, initially at room temperature and not permitted to exceed 120°F., circulating at the rate of 5 milliliters per second, shall be directed against the end plate of the housing from a one mm. capillary connected to a pump taking suction from the reservoir of water under the bearing so that it impinges on the end plate one-quarter of an inch above the outer opening of the bearing housing, as illustrated in Figure 2. Operation shall be continued for one hour when the bearing shall be removed, dried at 180°F. and reweighed to determine the grease loss. This grease loss divided by the weight of the grease used in packing the bearing shall be reported as the per cent grease loss."

Data obtained from laboratory evaluations of the above mentioned types, but particularly experience in the field, have indicated that the ability of greases to resist wash-out from bearings operated under service conditions is dependent on the following factors.

1. Metallic component of soap (calcium, sodium, etc.).
2. Fatty component of soap (oleate, stearate, etc.).
3. Mineral oil (viscosity, degree of refinement, etc.).
4. Reaction of finished grease (free acid or free alkali).
5. Additives (rust and oxidation inhibitors, etc.).
6. Bearing design (sealed, open, etc.).
7. Operating conditions (temperature, frequency of application of grease, etc.).

The samples used for the tests covered by Table I were prepared so that they differed essentially in only one respect, i. e., the alkaline base used for the saponification of the stearic acid. All of the greases included in Tables I and II were made with the same mineral oil having a viscosity of 105 seconds at 100°F.

**Table I**

**Effect of Metallic Component of Soap on Water Resistance of Greases**

Soap Content: 13-15%. Approx. Unworked and Worked Penetrations @ 77°F.: 250

| Grease Soap Base | AN-G-5a Procedure Grease Loss, % | Navy Water Absorption Procedure Water Absorbed, Per Cent |
|---|---|---|
| Calcium Stearate | Nil | 25 |
| Sodium Stearate | 25 | 35 |
| Aluminum Stearate | Nil | 20 |
| Lithium Stearate | 3 | 20 |

It will be observed that when tested by the so-called AN-G-5a procedure, the amount of wash-out of the calcium, aluminum and lithium stearate greases was quite small. The sodium stearate grease emulsified to a considerable extent and the loss of product from the bearing was appreciably higher. In the so-called Navy Water Absorption Test, the same three greases retained water to about the same extent with the sodium stearate product showing a somewhat higher percentage.

**Table II**

**Effect of Organic Component of Soap on Water Resistance of Greases**

Soap Content: 13-15%. Approx. Unworked and Worked Penetrations @ 77°F.: 250

| Grease Soap Base | AN-G-5a Procedure Grease Loss, % | Navy Water Absorption Procedure Water Absorbed Per Cent |
|---|---|---|
| Sodium Stearate | 25 | 35 |
| Sodium Stearate-Oleate (Stearic Acid-Oleic Acid Ratio 50-50) | 30 | 100+ |
| Sodium Oleate | 90 | 100+ |

It is often assumed that all sodium base greases will form emulsions which will flow like liquids from the bearings and leave no residue. Actually, wide differences have been noted with changes in composition of the greases. In Table II, sodium oleate grease showed much less resistance to water than the corresponding grease made with the soap of

Continued on following page

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a high titre fatty acid, i. e., stearic acid. In the Water Absorption Test, the sodium stearate grease acquired the consistency of a firm paste whereas the sodium oleate product was converted to a comparatively thin emulsion. In the case of other types of greases, water resistance is also determined to some extent by the chemical composition of the organic component but this factor is more readily demonstrated for sodium base lubricants.

**Table III**

**Effect of Mineral Oil Viscosity on Water Resistance of Greases**

Sodium Stearate Soap Content: 13-15%. Approx. Unworked and Worked Penetrations @ 77°F.: 250

| Approx. Saybolt Viscosity of Mid-Continent Mineral Oil at 100°F., Secs. | AN-G-5a Procedure Grease Loss, % | Navy Water Absorption Procedure Water Absorbed Per Cent |
|---|---|---|
| 100 | 25 | 35 |
| 1000 | 20 | 40 |
| 3000 | 8 | 100 |

The results of wash-out tests in Table III show that the tendency of sodium base greases to emulsify to form oil-in-water dispersions decreases as the viscosities of the mineral oils they contain increase. The water absorption results indicate that the quantities of water retained in the products increases as the viscosities of the mineral oils increase. There is good reason to believe that the water resistance properties are also affected by the degree of treating which the mineral oils have undergone. Refining of the oils results in removal of various natural components and their differences in this instance may be attributed to changes in chemical composition.

It may be of interest to offer a few comments regarding the interpretation of the results obtained by the Navy Water Absorption Method. The per cent water absorbed does not reliably indicate the extent to which the consistency of the grease has changed. Attempts[1] to correlate changes in worked penetrations with variations in amounts of water retained in the grease have not been particularly successful, because the changes in consistency during the test are due to the severe working to which the sample is subjected by the stirring device as well as to the effect of the water on the structure of the soap. Furthermore, the characteristics of the emulsions may vary considerably during the test as the rate of dispersion of the water is varied, particularly at the beginning of the run. As a rather unusual example, mention may be made of the test on a wheel bearing grease containing a mixture of sodium stearate and tallow soaps. The consistency of the emulsion dropped while 80% of water was dispersed in the sample and then a recovery of body occurred until about 200% of water had been added. Expressed in terms of micro penetrations obtained with a Texaco type penetrometer, this grease changed from an original of 58 to 346 at 20% water absorbed to 197 after about 200% water had been worked into the product. To further show the erratic behavior possible in this respect, a parallel test was run with the sample maintained in a flooded condition instead of adding the water in small increments. The sample then broke down to a creamy paste, too thin to check with a penetrometer.

1 Coordinating Research Council Report Project No. CLLG-13-43, "Water Resistance of Lubricating Greases."

Micro examinations of the soap structures of the greases before and after water has been dispersed therein often reveal interesting data. For example the inspection of a sponge grease made with soda tallow soap will disclose a tough, coarse, long fibred structure. After water is added and emulsification has occurred, micro examinations will show a change to a comparatively fine structure with the soap fibres quite short and thin. Similar slides prepared for lithium soap greases will show at 100 magnification, little apparent change in shape of the soap fibres but will disclose a more complete dispersion.


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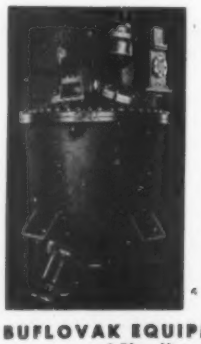

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The formation of emulsions of both types, i. e., oil-in-water and water-in-oil, and possibly compound emulsions, is of primary importance in determining the effect of additions of water. The amount of free alkali or free fatty acid present in the grease is therefore a factor as it may play a part in determining the adjustment of the stability of the emulsion. Rust inhibitors, such as emulsifiers and lead soaps, likewise affect ease of emulsification.

Under service conditions, the ability of the grease to stay put when the bearings are operated in contact with large amounts of water, is determined to an important extent by the design of the equipment. A well known example is car and truck wheel bearings where sodium base greases are normally employed without complaints from washout although the vehicles are used during rain and snow storms. Frequency of application is an important factor, particularly when the grease must be water repellent and function as a seal for the bearing and when a balance between maintenance of lubrication and rust prevention is dependent on adding new grease to make up for that washed out.

The seven factors listed above as determining the ability of a grease to resist wash-out from the bearings also affects its ability to protect the bearings from rusting when they are exposed to contact with water including condensation from humid atmospheres. However, experience has shown that if the grease is to satisfy both objectives, some compromise may be necessary because improved rust protective properties are usually obtained by favoring the factors which tend to encourage emulsification of the dispersed water. For example, it is well known that the incorporation of a sodium soap may appreciably improve the corrosion-prevention properties of conventional calcium base greases. It has been generally observed that the lubricant to be an effective rust preventative must contain a component which will preferentially wet the bearing surfaces in the presence of moisture and/or promote the formation of the required type of emulsion. On the other hand, if emulsification proceeds too rapidly to the point where the grease is not sufficiently retained in the bearings to provide adequate lubrication, then that grease is of questionable practical value for the application involved.

Continued on page 12

# TESTING ★ TOPICS ★

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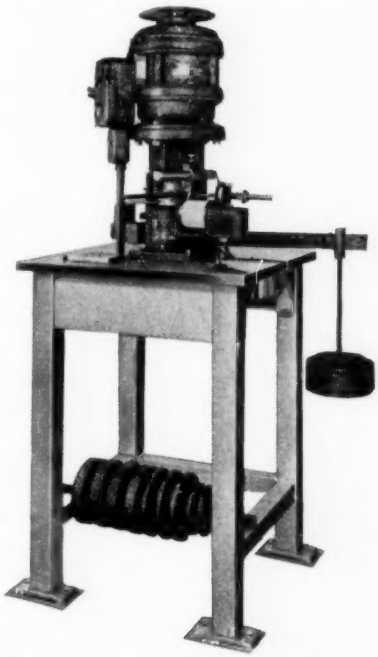

From the results of wear tests, it is possible to determine the most effective concentration of additives for reducing wear and friction. Wear tests on this machine have shown that oil rated high as E. P. Lubricants may differ considerably in regard to both wear and friction at moderate loads. Loads can be varied from 0.1 to 50 kilograms. Speed range 250 to 4,000 r.p.m. Temperature control 30 to 230° C.

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# A MICROWORKER *for* LUBRICATING GREASES*

• By GEORGE M. HAIN

*EDITOR'S NOTE: A set of the Microworker apparatus pictured here can be secured by addressing the author, Mr. Geo. M. Hain, 121 Park Street, Montclair, N. J.*

SYNOPSIS

A new instrument is described for mechanically working 5-g. samples of lubricating greases and for measuring the consistency of these greases at any stage of the working process. It consists of two 5-cu. cm. hypodermic syringes connected together tip to tip through a shearing element. The technique used for measuring consistency is the conventional micropenetrometer needle with a novel cup for holding the grease. Whereas the present 100,000-stroke test in an A.S.T.M. worker requires over 24 hr., the same information can be obtained on most greases in about 10 min. using the new microworker. This new method of testing greases greatly expedites the evaluation of mechanical stabilities and makes probable significant advances in the development and specification of greases.

A tentative method D217—44T[3] describes a method for measuring the consitency of petroleum greases. Part of this test requires that a sample of approximately one pound be worked 60 cycles in a mechanical worker which squeezes the grease back and forth through a plate perforated with ¼-inch holes. Sixty strokes was adopted because practice showed that most commonly manufactured greases did not soften much further with additional working. These greases were the conventional kettle-made greases which are manufactured by cutting back a stiff oil and soap paste with additional oil. The grease is paddled continuously and cooled somewhat during the cutback process. The mechanical worker needs only to break down the structure formed in the cooling which takes place after the grease is packaged.

During the war, however, lithium soap grease in large quantities was manufactured for the Army and Navy by the fire kettle procedure. The rigid gel formed on cooling without agitation was often only partially worked before packaging. Therefore, the standard 60-stroke penetration proved unreliable in predicting the performance of these greases in the bearings of high-speed motors. Army and Navy agencies have done considerable work in determining the mechanical stability or the extent of softening in greases when they are worked in the conventional worker to a million or more cycles. For much of this work a micropenetrometer suggested by Kaufman, Finn, and Harrington[4] had been used to measure the consistencies, but no instrument for working a small sample of the grease has been proposed. For much experimental work, especially in the field of synthetic fluid base lubricants, the size of sample required by the A.S.T.M. test, or modifications of it, is prohibitive. The A.S.T.M. test is also slow since only one test per day can be run in the automatic grease workers used in the 100,000-stroke test. The microworker described here permits much more rapid determinations on a 5-g. sample.

The new worker (see Fig. 1) consists of two 5-cu. cm. hypodermic syringes locked together tip to tip through a shearing element *E*. The shearing element consists of two cut-off hypodermic needle hubs soldered together with a fine mesh screen across the joint as shown in *D*. The lock-type syringe manufactured by Becton, Dickinson & Co. (Yale Lok 5YL) is recommended because the lock tip prevents tip breakage and makes a rigid assembly which cannot fly apart.

TABLE I.—EFFECT OF WORKING IN MICROWORKER

| | Micropenetrations after Following Number of Passes | | | |
|---|---|---|---|---|
| Grease | Unworked | 1 | 10 | 60 |
| | 250 Mesh Screen | | | |
| No. 3 | 52 | 54 | 53 | 60 |
| No. 4 | 86 | 92 | 92 | 96 |
| No. 5 | 59 | 67 | 126 | 200 |
| No. 2 | 90 | 110 | 176 | 300 |
| | 60 Mesh Screen | | | |
| No. 2 | 90 | 103 | 140 | 200 |

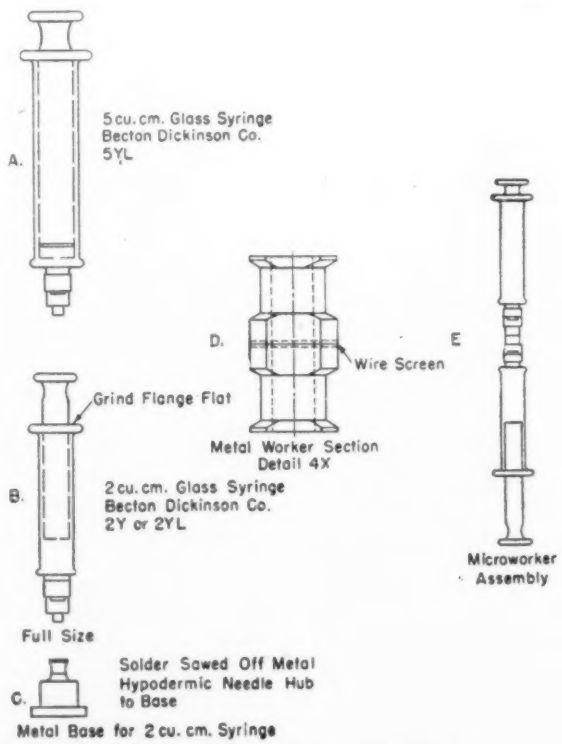


**Fig. 1.—Microworker for Greases.**

*Reprinted by permission from the ASTM Bulletin, August, 1947.

[4] G. Kaufman, W. D. Finn, and R. J. Harrington, "Miniature Penetrometer for Determining the Consistency of Lubricating Greases," *Industrial and Engineering Chemistry*, Anal. Ed., Vol. 11, p. 108 (1939). [This penetrometer is described in this issue of the ASTM Bulletin in the Appendix to the report by G. Kaufman on Micro Penetration of Lubricating Grease, see p. 81. ED.]

NOTE:—DISCUSSION OF THIS PAPER IS INVITED, either for publication or for the attention of the author. Address all communications to A.S.T.M. Headquarters, 1916 Race St., Philadelphia 3, Pa.

*Presented at a meeting of Section II on Consistency Measurements and Related Physical Tests for Lubricating Greases, of Technical Committee G on Lubricating Grease, of Committee D-2 on Petroleum Products and Lubricants, Atlantic City, June 16, 1947.

[1] The opinions or assertions in this paper are the author's and are not to be construed as official or reflecting the views of the Navy Department.

[2] Naval Research Laboratory, Washington, D. C.

[3] Tentative Method of Test for Consistency of Lubricating Grease and Petroleum (D 217-44 T), 1946 Book of A.S.T.M. Standards, Part II-A, p. 846.

when pressure is applied. The plunger is removed from one of the 5-cu. cm. syringes, and a solid plug of grease is pulled into it through the base by applying suction to the tip. The excess grease is then pushed out through the tip with the plunger, and the worker section and second syringe assembled as shown in Fig. 1-*E* with the plunger of the second syringe all the way down. The grease is pushed through the worker section into the second syringe. A No. 10 rubber stopper is a convenient support against which to press the plunger. The consistency of the grease after any number of passes through the screen is measured as follows: The tip of a 2-cu. cm. syringe (Fig. 1-*B*) is attached through an adapter to the tip of the 5-cu. cm. syringe, and the grease is forced in until the 2-cu. cm. syringe plunger is expelled. The 2-cu. cm. syringe is mounted in base *C* to measure the penetration. The Becton, Dickinson & Co. syringe 2Y or 2YL has internal dimensions nearly the same as the Kaufman micropenetrometer cup. The micropenetrometer cone is used for all consistency measurements. After the penetration is measured, the grease may be forced back into the 5-cu. cm. worker again. The syringe-to-syringe adapter is the same as the worker section *D* except that there is no screen between the needle hubs.

The object of the worker is to break down aggregates of flocculated individual soap crystals and also the continuous sponge-like structures of soap fibers. Large holes are unsuited to the latter process, although the shearing forces set up by flow through these holes is usually sufficient to break down loosely flocculated aggregates. When a wire screen is substituted for the perforated plate, each wire acts as a cheese cutter cutting through the rigid gel particles, and the shearing forces set up by flow through fine holes gradually breaks down these gel islands into individual fibers, a fact easily demonstrated by the dark field microscope. Fine screens are not possible in the conventional grease worker because the pressure per unit area is not high enough. With hypodermic syringes a pressure of 150 psi. is easily attained.

With a 400-mesh screen (0.0010-in. diameter wire) the microworker is suitable for lump-free greases as stiff as 240

*Continued on following page*

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# A MICROWORKER FOR LUBRICATING GREASE

Continued from page 9

A.S.T.M. penetration. However, when greases as stiff as these are worked, the screen should be examined frequently and replaced as soon as one or more of the wires break at the edge in fatigue failure. Monel or corrosion-resistant steel wire mesh in the range of 60 to 250 meshes per inch is suitable for greases stiffer than 240 A.S.T.M. penetration. The finest screen commercially available is 400 mesh screen from the Newark Wire Cloth Co. Although 1000 by 120 mesh woven cloth is also available, the open area is so much smaller in proportion to the total plate area that lumps tend to be held back until a solid cake is formed. For still greases, syringes of smaller bore size permit higher working pressures. Screens of mesh as coarse as 60 are useful for very stiff greases since the pressure required is considerably less than for a 250 or 400 mesh screen.

The microworker works the grease in a sealed system so that no air is drawn in during the process. Another advantage lies in the small cross-section, so that the whole apparatus can be placed for a few minutes in a water bath to bring the grease to the correct testing temperature. This has been a tedious process with the standard worker because of the slow heat transfer through a large mass of grease.

Table I gives the results obtained with the microworker. In Table II are given for comparison the measurements on the same greases obtained at the Engineering Experimental Station, Annapolis, Md., using 100,000 strokes in the A.S.T.M. worker in terms of A.S.T.M. penetrations. The data of Tables I and II show that the microworker evaluates the greases in the same order as the tedious standard procedure. The micropenetrations are difficult to correlate with A.S.T.M. penetrations when different types

TABLE II.—EFFECT OF 100,000 STROKES IN A.S.T.M. WORKER (SAME GREASES AS IN TABLE I).

| Grease | A.S.T.M. Penetrations after Following Number of Passes: 60 ¼-in. Holes | 100,000 ¼-in. Holes | 100,000 ⅛-in. Holes |
|---|---|---|---|
| No. 3 | 188 | 238 | ..... |
| No. 4 | 266 | 363 | ..... |
| No. 5 | 290 | 370 | 400+ |
| No. 2 | 313 | 400+ | ..... |

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of greases are involved, so no attempt has been made to convert between the two systems.

Data obtained on lithium soap diester greases[3] show that the breakdown after 1 to 500 passes through a 400 mesh screen is permanent, even after so long a waiting period as 6 weeks. There is some apparent recovery if the Kaufman split cup is used for the penetration test, but this disappears when the grease is agitated in transferring it from a large syringe to the 2-cu. cm. measuring syringe.

The greatest contribution of the microworker is in showing the effect of rust inhibitors, free acidity, or other polar impurities on the work stability of a grease. In Table III all the greases have the same soap content and substantially the same fluid base except for additives and impurities. The fluid in all cases is di-2-ethyl-hexyl sebacate, and the soap is lithium stearate. Grease A was made from the crude diester which had been freed of polar impurities by absorption methods. Grease B was made from the same purified diester, but contained 2 per cent of sorbitan monoleate as a rust inhibitor. Grease C was made from the crude diester but was otherwise identical with grease A.

TABLE III.—USE OF MICROWORKER TO EVALUATE THE EFFECT OF ADDITIVES ON SHEAR STABILITY.

| Grease | Micropenetrations after Following Number of Passes Through 400 Mesh Screen | | | |
|---|---|---|---|---|
| | Unworked | 1 | 10 | 100 |
| No. A | 74 | 81 | 89 | 100 |
| No. B | 116 | 142 | 177 | 214 |
| No. C | 125 | 135 | 164 | 230 |

Note:—In processing, these greases had already been passed eleven times through a 70 by 80 mesh wire cloth.

In addition, the grease worker has many experimental uses because of the intense mixing it accomplishes. It is, in effect, a miniature mixing kettle. It can be used to test the effect of small quantities of additives such as water, antioxidants, or rust inhibitors on experimental greases. The device is useful for making experimental cutbacks to determine the effect of soap content on consistency and for making grease dispersions of the high dilutions necessary for microscopic examinations of grease structure under dark field conditions.

### Conclusions

The new microworker is a valuable device for the development and evaluation of greases because it permits rapid determination of the relative work softening of a grease on a very small sample. Results may be obtained in a few hours on a large number of samples which would take weeks to evaluate by any other known procedure. Its ability to mix the additives and filter the greases should also prove useful.

[3] G. M. Hain, D. T. Jones, R. L. Merker, and W. A. Zisman, "Synthetic Low-Temperature Greases from Aliphatic Diesters," *Industrial and Engineering Chemistry*, Vol. 39, p. 500 (1947).

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## *Evaluation of* Water Resistance Properties *of Lubricating Greases*

Continued from page 7

The evaluation of the rust protective properties of lubricating greases has generally followed the line of attack used for straight rust preventive compounds. I. A. Levin[2] has proposed a method wherein metal balls are coated as prescribed with films of grease samples and the "porosity" of coatings, submerged in a 0.001% NaCl solution, is determined by measuring the current conducted through the films. The minimum thickness of grease film required to assure freedom from "pores" is determined from curves. It is probable that during the past few years the method described in Navy Ordnance Specification OS 1350 and Army Ordnance Specification AXS-781, Revision 3, has received the most widespread attention. In this test, carefully prepared steel panels are given a coating of grease and exposed in a cabinet for 100 hours to a controlled spray of 4% synthetic sea water solution at a temperature of 90°F.-100°F. Greases are rejected if there is any evidence of corrosion of the metal when cleaned at the end of the test. It is important to note that the film of grease on the panels is extremely thin in this test. In fact, it is intended that the amount of coating be equivalent to that which would be formed by a medium viscosity oil after a lengthy drain period.

2 I. A. Levin—Korroziya i Borba s Nei, 6, 3, 1940

**Table IV**

**OS-1350 Salt Spray Test**

**4% Synthetic Sea Water**

| Grease Soap Base | Type Product | Hrs. Until Failure |
|---|---|---|
| Calcium Tallow | Army 2-107 | 4 |
| Sodium Tallow | Army 2-108 | 4 |
| Aluminum Stearate | Army 2-107 | 4 |
| Lithium Stearate | AN-G-3a | 4 |
| Lithium Stearate plus Rust Inhibitors | O. D. No. 00 | 150 |

The data covered by Table IV indicate the degree of improvement in rust preventive properties that can be attained by the use of properly selected additives when the products are evaluated by the above outlined procedure.

Resistance to corrosion from sea water is an important requirement, particularly for marine applications. However, experience has shown that the factor of safety potentially available with a rust inhibited grease may also be secured by the use of other more conventional greases when they are properly applied and thicker films are maintained.

Attention has been given to the adaptation of humidity cabinets for evaluation of rust protective properties of greases. Table V covers the results of tests wherein open 204 ball bearings packed two-thirds full with the grease samples were substituted for grease coated SAE 1020 panels. With the humidity maintained at the level of 100%, the test was to a considerable extent also an evaluation of the water repellency and resistance to wash-out properties as well as rust preventive characteristics. The results again indicate that the addition of a rust inhibitor to lithium stearate greases was effective. The sodium soap grease in this case, where the bearings were not rotated except for inspection purposes, failed to protect the bearings from rusting and the lubricant was washed out to the point where considerable areas were left un-

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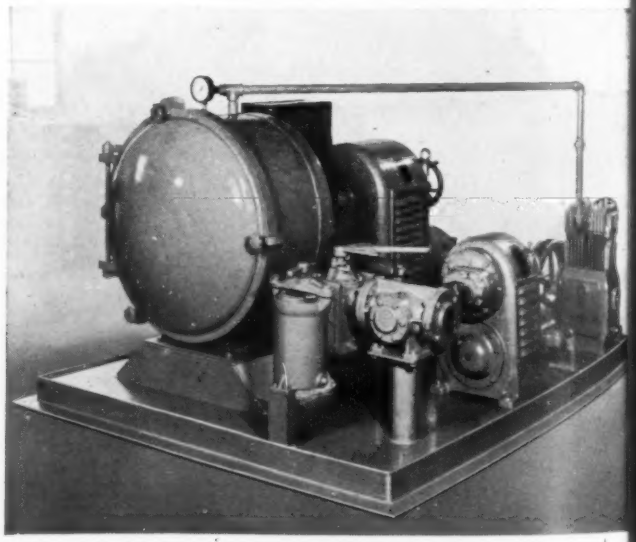

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**Table V**
**Humidity Cabinet Tests**
**100% Humidity @ 120° F.**
**No. 204 Ball Bearing Packed Two-thirds Full**

| Grease Soap Base | Type Product | Condition of Bearing After | | |
|---|---|---|---|---|
| | | 1 Month | 3 Months | 6 Months |
| Calcium Tallow | Army 2-107 | Difficult to start. No visible rust. | Rough turning. No visible rust. | Rough turning. No visible rust. |
| Sodium Tallow | Army 2-108 | Rough turning. Most of grease gone. Considerable rust. | Rough turning. Grease completely gone. Considerable rust. | Rough turning. Grease completely gone. Considerable rust. |
| Aluminum Stearate | Army 2-107 | Slightly rough turning. No visible rust. | Difficult to start. No visible rust. | Difficult to start. No visible rust. |
| Lithium Stearate | AN-G-3a | Slightly rough turning. No visible rust. | Difficult to start. No visible rust. | Difficult to start. No visible rust. |
| Lithium Stearate plus Rust Inhibitor | O. D. No. 00 | OK in all respects. | OK in all respects. | OK in all respects. |

protected by even a separated oil film. The bearings packed with calcium, aluminum and lithium soap greases were reported as showing no visible rust, or in other words, no rust that could be definitely detected without disassembling the bearings. Because the bearings were found to be rough turning, they were taken apart at the end of the tests. Close inspection of the balls and races showed corrosion had actually occurred at points of contact of the balls with the races and that this was the cause for rough turning of the bearings during the tests.

The data included in Table VI were obtained from another series of tests wherein coated panels were subjected to the different types of exposure noted. The tests conducted outdoors were kept on the roof of a laboratory building located in an area surrounded by manufacturing plants of various types. It is interesting to note that the only failure in the outdoor series was the panel coated

Continued on page 16

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. . . . . Mr. Stingley has worked with Fats, Oils and Fatty Acids and also Chemicals from Fats, both in the laboratory and in practical field application work. He has published a number of papers dealing with fatty acids.

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has been active in directing the develop ment and manufacture of items of in terest to the Petroleum industry, suc as metallic soap, organometallic com pounds and various metal reacti products.

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Mr. Vagtborg is a graduate of the Uniersity of Illinois, holds a degree from e Armour Institute of Technology and ark College (honorary). He was Direcor of Armour Research Foundation of linois Institute of Technology, 1937-45. irector and Organizer of the Institute Gas Technology, Chicago, 1941-43. resident and Director of Midwest Rearch Institute, Kansas City, 1945. Adinistered confidential war research ork carried on at Armour Research oundation for Office of Scientific Reearch and Development, Office of Proaction Research and Development, rmy and Navy. Recipient of certifiate from Office of Scientific Research d Development for outstanding conibution to successful prosecution of the

*Harold Vagtborg*

cond World War. Recipient of the ver Medal, Royal Swedish Academy of chnical Science. Published many arcles concerning industrial research, scitific research, institutions and research anagement as well as numerous techcal papers in journals of the National ientific Society.

. H. D. Hass, Head
partment of Chemistry
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fayette, Indiana
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Graduate of Ohio Wesleyan magna m laude at 19. Received his Ph.D m Ohio State four years later. Reech Director of the Baltimore Gas gineering Corporation, which position

*H. B. Hass*

carried him to the natural gas field where the sight of millions of tons of cubic feet of unused natural gas being blown into the air was profoundly disturbing to him. His major research efforts have been devoted to the chemical utilization of the hydrocarbons of natural gas and petroleum. In 1928, Dr. Hass went to Purdue as Assistant Professor and in a few months had inaugurated a research program which was destined to result in the establishment of a new industry based upon the vapor-phase nitration of gaseous paraffins. Promotion at Purdue came rapidly and in 1937 Dr. Hass was made head of the Department of Chemistry where a staff of approximately 273 imposes considerable administrative responsibility. In normal times the classes in general chemistry alone total 6,465 students.

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*Carl W. Georgi, Chairman NLGI Technical Committee*

**Agenda for the N.L.G.I. Technical Committee Meeting 1:30 P.M. Friday, October 17th**

Reports on Activities of Other Grease Committees:

(a) A.S.T.M. Technical Committee G
Section I on Analysis Methods
Gus Kaufman
Section II on Consistency Test Methods
E. W. Adams
Section III on Performance Tests
T. G. Roehner

(b) A.B.E.C.-N.L.G.I. Joint Committee
T. G. Roehner

(c) A.P.I. Lubrication Committee
Chassis Lubrication Panel
C. W. Georgi

*Continued on page 18*

**TECHNICAL COMMITTEE MEETING — 1:30 P.M., FRIDAY, OCTOBER 17TH**

**Table VI**
**Corrosion Tests on Representative Type Greases**
**2″ x 3″ Low Carbon Steel Panels**

| Grease Soap Base | Type Product | Outdoor Shed Test* | Outdoor Exposure Test** | Humidity Cabinet Test*** | Sea Water Immersion Test**** |
|---|---|---|---|---|---|
| Calcium Tallow | Army 2-107 | OK 6 Months | OK 6 Months | OK- 1 Month | Failed after 21 days |
| Sodium Tallow | Army 2-108 | OK 6 Months | Failed after 5 Months | Failed after 1 Month | Failed after 7 days |
| Aluminum Stearate | Army 2-107 | OK 6 Months | OK 6 Months | OK- 1 Month | OK- 1 Month |
| Lithium Stearate | AN-G-3a | OK 6 Months | OK 6 Months | OK- 1 Month | Failed in 8 days |

* Protected from rain and snow.
** Plates mounted on Panel at 40° angle facing south. No protection from rain and snow.
*** 100% Relative Humidity at 100°F.
**** Plates immersed in synthetic sea water.

Continued from Page 13

with an Army Specification 2-108 type wheel bearing grease and that the failure was preceded by removal of the grease by direct exposure to rain and snow. That same grease failed in the series run in the humidity cabinet because of excessive wash-off. In respect to the observations based on the immersion tests in synthetic sea water, it should be mentioned that the aluminum base grease employed contained an adhesive agent and a mineral oil having a viscosity of about 4000 seconds at 100°F.

In conclusion, the data and comments presented herein are the basis for the statement that while resistance of greases to wash-out from bearings and their rust preventive properties are interrelated, it is advisable to recognize that they represent different phases of the main subject of water resistance properties of lubricating greases. Recommendations based solely on the ability of the lubricants to stay put in the presence of

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water may not provide products which will also give good protection to the bearing from rusting. The data furthermore indicate the many factors which influence these desirable properties and which complicate development of laboratory methods for their evaluation. Experience under service conditions is usually more desirable than laboratory data and, in this case, it is particularly advisable to rely on information obtained in the field. A review of technical periodicals will disclose that there have not been many articles published on this subject. The Coordinating Research Council's Report, entitled "Amphibian Wheel-Bearing Grease (Project CLLG-37-44)," is a noteworthy example describing experience in the laboratory and field. It showed that good stay put properties had definitely limited benefits if rusting proceeded to the point where the bearings were no longer operable. Other case histories could be given where the reverse was true. Development and standardization of laboratory methods which will reliably predict field performance should therefore be encouraged.

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## 15th Annual Convention

### Agenda for the N.L.G.I. Technical Committee

Continued from page 15

Need for Less Expensive and Less Complicated Grease Test Apparatus and Methods:
- (a) Microworker
- (b) Simplified Anti-Friction Bearing Test Apparatus
- (c) Other Apparatus and Methods

New Institute of Petroleum Test Method for "Oil Separation on Storage."

N.L.G.I. Consistency Classification Proposals to request adoption by S.A.E. and/or A.S.T.M.

New Business

SYMPOSIUM ON PUMPABILITY OF GREASE AND DELIVERY CHARACTERISTICS OF DISPENSING EQUIPMENT:

(Continuation of October, 1946, Symposium on same subject)

Discussion and decision on future program and activity, particularly whether it would be desirable to set up a co-operative program to evaluate the pumpability characteristics of various representative types of greases and the delivery characteristics of typical types and models of dispensing equipment.

## TENTATIVE PROGRAM
### *of the*
## NATIONAL LUBRICATING GREASE INSTITUTE

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**THURSDAY, OCTOBER 16, 1947**

Registration—Foyer—8:30 A. M.
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**Morning Session—East Room**
10 A. M. to 12:30 P. M.

***Address of Welcome***
MR. H. P. HOBART, President, N. L. G. I.
Gulf Oil Corp., Pittsburgh

***"Fats, Oils and Fatty Acids for Industrial Purposes"***
MR. DALE V. STINGLEY
Armour & Co., Chicago, Ill.

***"Metallic Soaps"***
MR. S. B. ELLIOTT, Ferro Chemical Corp.
Bedford, Ohio

***Board of Directors Meeting***
12:30 P. M.

**Afternoon Session**
2:00 P. M. to 4:30 P. M.

***"Modernization of Grease Manufacturing"***
MR. C. L. JOHNSON, Pres. Jesco Lubricant Co.
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*The Grease Phase of Steel Plant Lubrication*
Mr. C. E. Pritchard, Chief Lubrication Engineer
Republic Steel Corp., Cleveland, Ohio

*Annual Business Meeting*
4:30 P. M.

## FRIDAY, OCTOBER 17, 1947

### Morning Session

10:00 A. M. to 12:30 P. M.

*"Research—The Third Dimension"*
Mr. Harold Vagtborg, Pres. Midwest Research Institute
Kansas City, Mo.

•

### Friday Afternoon

2:00 P. M.—4:00 P. M.

*N. L. G. I. Technical Committee Meeting*
Mr. Carl W. Georgi, Chairman
Enterprise Oil Co., Buffalo, Presiding

*Reports on Activities of Other Grease Committees:*

(a) A.S.T.M. Technical Committee G
Section I on Analysis Methods
Gus Kaufman
Section II on Consistency Test Methods
E. W. Adams
Section III on Performance Tests
T. G. Roehner

(b) A.B.E.C.—N.L.G.I. Joint Committee
T. G. Roehner

(c) A.P.I. Lubrication Committee
Chassis Lubrication Panel
C. W. Georgi

*Need for Less Expensive and Less Complicated Grease Test Apparatus and Methods:*

(a) Microworker

(b) Simplified Anti-Friction Bearing Test Apparatus

(c) Other Apparatus and Methods

*New Institute of Petroleum Test Method for "Oil Separation on Storage."*

*N. L. G. I. Consistency Classification Proposals to request adoption by S.A.E. and/or A.S.T.M.*

*New Business.*

*Symposium on Pumpability of Grease and Delivery Characteristics of Dispensing Equipment:*

(Continuation of October 1946 Symposium on same subject)

Discussion and decision on future program and activity, particularly whether it would be desirable to set up a cooperative program to evaluate the pumpability characteristics of various representative types of greases and the delivery characteristics of typical types of models of dispensing equipment.

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### COCKTAIL HOUR

6:00 P. M.—West Lounge
Members and their guests, including ladies

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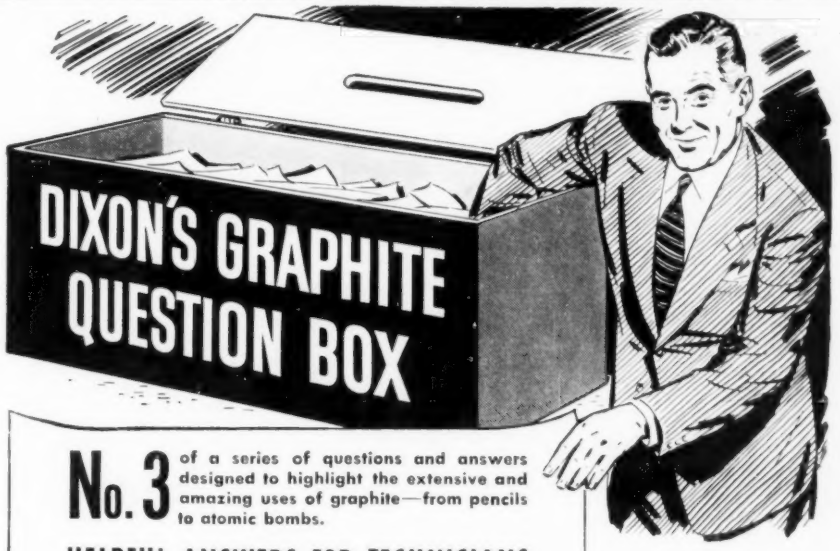
### ANNUAL BANQUET

7:00 P. M.—Grand Ball Room


**No. 3** of a series of questions and answers designed to highlight the extensive and amazing uses of graphite—from pencils to atomic bombs.

**HELPFUL ANSWERS FOR TECHNICIANS AND PRODUCT ENGINEERS**

**QUES.** Is graphite processed into all required forms and shapes that make it usable for innumerable manual, mechanical, chemical and electrical applications?

**ANS.** Graphite owes its remarkable versatility to its almost unlimited scope of form and formulation. It is available in a wide variety of microscopic powders, large flakes, fluids, non-fluids, concentrates, bars, tubes, sheets, plates and specially processed shapes.

**QUES.** What are *some* of the products or processes in or on which graphite is used? (*Continued from No. 2*)

**ANS.** Suspensions and concentrates
Pressure pad lubricant for belt type sanding machines
Penetrating and rust dissolving lubricants
Stop cock lubricants
House movers' lubricant
Lace machine lubricant
Boiler water-scale conditioner
Miscellaneous grease, oil and aqueous lubricants
Miscellaneous grease and oil compounders' graphites
Miscellaneous lubricants of special structures, densities and shapes
Gunpowder glazing and pelleting
Glass makers' lubricants
Ferry rack lubricants
Synthetic rubber tire tube lubricant
Auto under-chassis spraying lubricant
Auto spring and shackle lubricant
Miscellaneous marine engine and equipment lubricants
Miscellaneous aircraft lubricants

*On Railroads:*
Brake cylinder lubricant
Triple valve lubricant
Curved track lubricant
Centerplate lubricant
Diaphragm, chafing plate and pedestal plate lubricant
Pantograph shoes
Hub liner lubricant
Driving spring lubricant
Cup and driving journal lubricant
Semaphore lubricant
Car seat and window slide lubricant
Engine front and jacket finishes

(*To be continued in No. 4*)

A few of thousands of widely used products containing Dixon's Graphites. Those starred are Dixon's products, many of them sold by supply houses everywhere.

★ Slipstik, Greastik, G-711 Stick

Brake Linings

★Cup Grease No. 3

Now may we receive *your questions* on how graphite can help *you*?

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